

Virginia Division of Consolidated Laboratory Services

SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION by EPA 3510C REVISION 3 (1996)					
Facility Name: _____ VELAP ID _____					
Assessor Name: _____ Analyst Name: _____ Inspection Date _____					
Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst: _____					
Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____					
Did each analyst demonstrate his/her ability to produce acceptable results with this method?	1.3				
Were acceptable grades of reagents used for this method?	5.0				
Was Sodium Sulfate purified by heating to 400°C for 4 hours or precleaning with methylene chloride?	5.4				
Were sample pH's adjusted according to Table 1?	7.3				
Were sample volumes measured with a graduated cylinder or the sample containers marked for volume determination prior to placing samples into separatory funnels?	7.1				
Were volumes of surrogate and/or matrix spiking solution placed into sample prior to extraction?	7.2				
If method 3640, Gel-Permeation Cleanup was employed, were twice the necessary volumes of surrogate or matrix spike solutions added, as half of the extract will not be recovered?	7.2.2				
If graduated cylinders were used to measure sample volumes, were they rinsed with 60 mL methylene chloride after transfer?	7.4				
If the samples were placed directly into separatory funnels, were the sample containers rinsed with 60 mL methylene chloride after transfer?	7.4				
Were separatory funnels shaken vigorously for 1-2 minutes with frequent venting after methylene chloride addition?	7.5				
Were organic and aqueous phases allowed to separate for at least 10 minutes?	7.6				
If emulsions were formed that were greater than one-third the size of the solvent layer, were mechanical emulsion-breaking techniques employed?	7.6				
Notes/Comments:					

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If emulsion could not be broken so that more than 80% of the methylene chloride could be recovered, was method 3520, continuous liquid-liquid extraction used?	7.6				
Was extraction process repeated twice more?	7.7				
Was extraction process repeated more times if necessary?	7.8				
Were acid/neutral and base extracts combined or analyzed separately according to whichever determinative technique was used?	7.9				
<b>Kuderna-Danish (K-D) Concentration Technique</b>					
Were K-D concentrations performed on extracts if necessary?	7.10				
Were extracts first dried by passing them through 10 cm of anhydrous sodium sulfate?	7.11.3				
If other solvents were necessary for determinative techniques, was methylene chloride exchanged for those solvents?	7.11.6				
<b>Micro-Snyder Column Technique</b>					
If further concentration was indicated by Table 1, were extracts further concentrated to 0.2 mL and brought to 1 mL again with methylene chloride or exchange solvent?	7.12.1				
<b>Nitrogen Blowdown Technique</b>					
Were concentrator tubes placed in a warm bath of about 35°C, and the solvents evaporated to the volumes indicated in Table 1 with a gentle stream of clean nitrogen?	7.12.2.1				
Were the internal walls of the tubes rinsed several times with solvent?	7.12.2.2				
Were extracts <b>not</b> evaporated to dryness?	7.12.2.2				
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<b>Quality Control</b>					
If analysis was not to be performed immediately, were concentrator tubes capped and stored under refrigeration?	7.13				
If extracts were to be stored for longer than 2 days, were they transferred to vials with PTFE-lined caps and labeled?	7.13				
Were all reagent blanks, matrix spikes, and replicates subjected to the same processes as samples?	8.1				
Notes/Comments:					

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TABLE 1  
SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Deter- minative method	Initial extraction pH	Secondary extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for cleanup (mL)	Final extract volume for analysis (mL) <sup>a</sup>
8041	≤2	none	2-propanol	hexane	1.0	1.0, 0.5 <sup>b</sup>
8061	5-7	none	hexane	hexane	2.0	10.0
8070	as received	none	methanol	methylene chloride	2.0	10.0
8081	5-9	none	hexane	hexane	10.0	10.0
8082	5-9	none	hexane	hexane	10.0	10.0
8091	5-9	none	hexane	hexane	2.0	1.0
8100	as received	none	none	cyclohexane	2.0	1.0
8111	as received	none	hexane	hexane	2.0	10.0
8121	as received	none	hexane	hexane	2.0	1.0
8141	as received	none	hexane	hexane	10.0	10.0
8270 <sup>c,d</sup>	<2	>11	none	-	-	1.0
8310	as received	none	acetonitrile	-	-	1.0
8321	as received	none	methanol	-	-	1.0
8325	7.0	none	methanol	-	-	1.0
8410	as received	none	methylene chloride	methylene chloride	10.0	0.0 (dry)



METHOD 3510C  
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION

